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Photochromic organic compounds for optical memory

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For the purpose of target syntheses of new photochromic compounds, a relationship between the structure and properties of a series of the known thermally irreversible photochromes was analyzed using the spectral kinetic data obtained by the authors.

Key words: photochromism, optical memory, diarylethenes, fulgimides.

Progress in information technologies demands a sharp increase in the informational capacity of information carriers and in the rate of data processing.

An analysis of the modern state of research in this area[1] shows that the limiting density of information recording on magnetic discs can reach 230 Gbit inch^{-2}. In this case, the informational capacity of magnetic discs can be increased to 20 Gbit. The rate of data processing can be 1 Gbit s^{-1}.

In addition to magnetic discs, optical discs (ODs) for archive storage of information are widely popular. They can be of the CD type (type I), providing multiple reading of an optical information recorded in a studio, and of the WORM type (type II) with a single recording and multiple reading directly in a personal computer. Reversive ODs of the WERM type (type III) were created for operative multiple recording and repeated recording of optical information and its multiple reading. Optical information recording on the most part of known ODs is based on thermoinduced processes of substance conversion. In the case of ODs of types I and II, information recording is performed due to the thermoinduced ablation of deposited *in vacuo* layers of organic and inorganic substances. Reversible recording — rewriting of optical information in an OD of type III occurs due to the thermoinduced magnetization reversal of magnetic domains in optical discs or reversible phase transformations of metallic alloys. The informational capacity of these ODs used for information reading from a semiconducting laser with a radiation wavelength of 650 nm is ~4.7 Gbit. New commercial ODs developed by the Blu-ray Disc Association have the record-breaking informational capacity: 25 Gbit for single-sided and 50 Gbit for double-sided (DVD) information carriers, which is achieved by the application of a semiconducting laser with a shorter wavelength (405 nm), an optical system with a digital aperture of 0.7/0.8, and the photosensitive layer with a thickness of 0.1 mm.

Prospects for increasing the informational capacity of ODs are related to the creation of multilayer photosensitive coatings for bitwise information detection and thick (>2 mm) photosensitive layers for holographic information detection. These ODs provide the three-dimensional (3D) optical memory with the recording density higher than 1 Tbit due to information recording and reproduction in various layers of a detecting medium (in the case of the bitwise optical memory) or at different incident angles of the activating and recovering laser radiation (in the case of the holographic optical memory).

The prospects for improving ODs for optical memory are associated with the use of photosensitive organic media that experience photochemical transformations. In principle, these media possess a higher resolving ability than the presently used thermosensitive materials.

The purpose of the present study is the search for thermally irreversible photochromic organic compounds with two thermodynamically stable states for the development of photochromic detecting media that provide the creation of multilayer optical discs (Fig. 1). The phenomenon of two-photon photochromism is used to create the 3D bitwise operative optical memory (WERM)[2] based on the two-photon ODs.[3]

Photochromism is the ability of a substance to reversibly change (under optical irradiation) the visible absorption spectrum, *i.e.*, the color and other properties caused by the formation, in essence, of a different compound.[4]

During the photochromic process, a substance absorbs optical radiation and transits from the initial state **A** to the so-called photoinduced state **B** characterized by a

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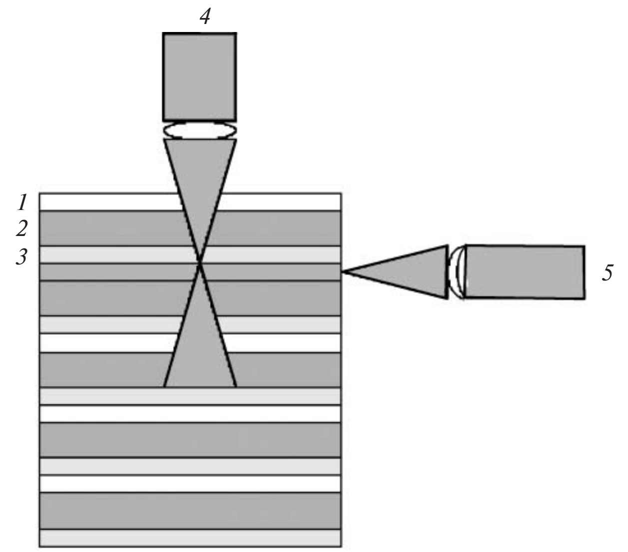


Fig. 1. Scheme of the multilayer detecting medium consisting of consecutive layers: detecting (*1*), address (*2*), and isolating (*3*), as well as the scheme of recording using one (*4*) or two beams (*4* and *5*) of the same laser.

different absorption spectrum and a certain lifetime. The backward transition from state **B** to state **A** occurs spontaneously due to the internal energy and can considerably be accelerated on heating of the substance or under the action of the light absorbed by form **B**.

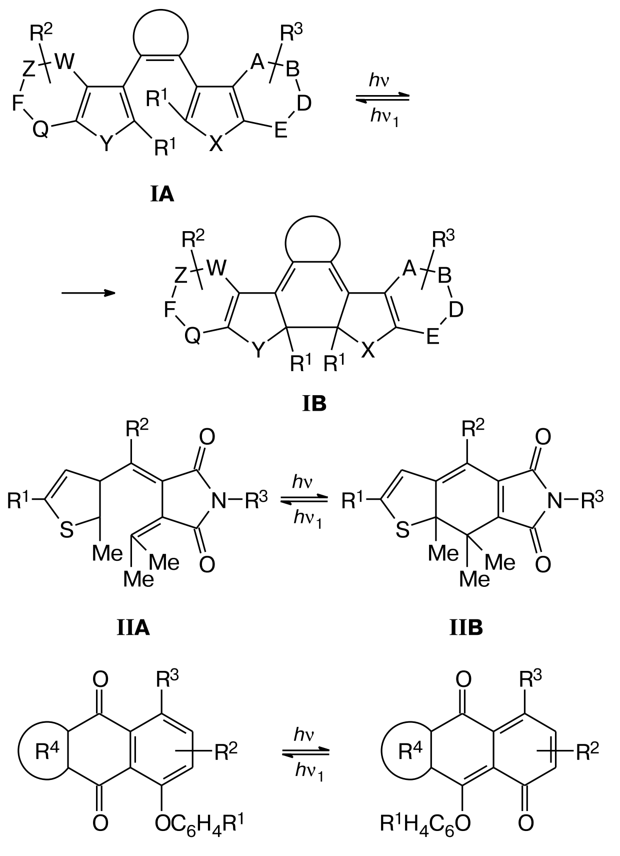
$$\mathbf{A} \underset{h\nu_2,\ \Delta}{\overset{h\nu_1}{\rightleftharpoons}} \mathbf{B}$$

The photochromism phenomenon is inherent in many organic and inorganic substances. Photochromism is based on both the photophysical processes (triplet—triplet absorption, singlet—singlet clarification) and numerous photochemical reactions accompanied by the rearrangement of covalent bonds (during photodissociation, redox photochemical reactions) or a change in the configuration of molecules (*cis—trans*-isomerization).[4—6] Several materials for use in various areas of science and technology have been developed on the basis of the organic and inorganic photochromic substances.[4—7]

For some types of photochromes, the thermal relaxation of photoinduced form **B** becomes difficult because of high thermodynamic barriers for the transition from the ground state of form **B** to the ground state of form **A**. Such photochromic compounds were named thermally irreversible but photochemically reversible.[8] Just these products with two thermodynamically stable states are appropriate for the creation of photochromic detecting media for the operative 3D optical memory. In addition, photochromes for these purposes should possess a low efficiency of photodegradation, thus providing a high fatigue resistance in optical information recording—repeated recording and the two-photon absorption coefficient and, hence, can provide non-destructive optical information reading.[9] These photochromic compounds are few and mainly related to the classes of diarylethenes **I**,[10] fulgides and fulgimides **II**,[11] and phenoxy-substituted derivatives of quinones **III**.[12]

All compounds of the indicated types are subjected to reversible photoinduced valence isomerization. Diarylethenes **I** and fulgimides **II** undergo reversible phototransformations between the opened and cyclic forms, and the phenoxy-substituted derivatives of quinones **III** are characterized by reversible phototransformations between the *ana*- and *para*-quinoid forms (Scheme 1).

Scheme 1



In this work, we analyze the spectral kinetic properties of the recently synthesized thermally irreversible photochromic compounds belonging to the classes of diarylethenes and fulgimides.

Experimental

Photochromic diarylethenes[13—30] and fulgimides[23—31] used in the work were synthesized according to known procedures.

Investigation was performed in solutions. The solvent was toluene (A.C.S. spectrophotometric grade). The concentration of the compounds in solution was $2 \cdot 10^{-4}$ mol L^{-1}. Measurements were carried out in a cell 3 mm thick.

Absorption spectra of the opened and cyclic forms of the photochromes were recorded on a Shimadzu UV—VIS spectrophotometer in a spectral range of 200—800 nm. The colored cyclic form was obtained after the solution was photoexcited with the radiation from a DRSh-250 mercury lamp passed through the UV light filter that picked out the radiation with $\lambda = 313$ nm. The kinetics of photocolorization of solutions of diarylethenes was measured at the wavelength of the absorption band maximum of the cyclic form appeared upon the UV irradiation of solutions of these compounds preliminarily decolorized by the visible light.

The photodecolorization kinetics was studied upon the irradiation of preliminarily colored solutions of these compounds by the radiation of mercury lines from a DRSh-250 lamp in the visible spectral region, which were picked out by glass filters that transmitted the radiation in the region of the absorption band of the cyclic form. The kinetics of photocolorization and photodecolorization processes before the beginning of efficient photodegradation was the first-order reaction kinetics, because reversible phototransformations occurred only between the opened and cyclic forms of diarylethenes.

The kinetic curves of photodecomposition of the photochromic compounds were used for the accelerated estimation of their fatigue resistance in the photochromic transformations under the conditions of high-intensity irradiation. The photodecomposition of the solutions was characterized by the time during which the absorbance achieved in the photostationary state at the absorption band maximum of the cyclic form halved upon continuous irradiation with the non-filtered light from a DRSh-250 lamp.

The kinetics of thermal isomerization of the photoinduced form of diarylethenes and fulgimides was studied by periodical (at an interval of several days) measurement of absorption spectra of solutions. In intervals of the measurements, the solutions were stored in dark.

All kinetic studies were carried out under the same experimental conditions at comparable intensities of the incident UV and visible radiation, which provided a possibility of comparative analysis of the obtained results.

Special attention was given to the stability of the compounds to irreversible phototransformation (photodegradation), which results in their fatigue resistance in photochromic transformations between the opened and cyclic forms during optical information recording—repeated recording. In addition, a correspondence between the absorption band maxima of the both forms and wavelengths of the existing lasers was determined. The photoinduced absorbance in the absorption band maximum for the cyclic form in the equilibrium state upon UV irradiation was also measured, which made it possible to estimate a comparable photosensitivity of the compounds to the recording UV radiation. The ratio of the photocoloration and photodecoloration rate constants was used for the comparable estimation of the efficiency of these processes.

Results and Discussion

The objects of the study were dihetarylethenes **1—39** with various bridging fragments, as well as fulgimides **40—44** and bisfulgimides **45—49**.

Diarylethenes. The preliminary studies show that all the synthesized photochromic diarylethenes are thermally irreversible, because the storage of their irradiated solutions in dark for one month produced no substantial changes in the UV-induced absorbance in the absorption band maximum of the cyclic form.

The results of a comparative spectral kinetic study of diarylethenes **1—21** with similar structures of the thienyl moieties but with different bridging fragments of 15 types are presented in Table 1.

As can be seen from the data in Table 1, perfluorocyclopentenes **1** and **17** and cyclopentene **18** possess the highest stability toward irreversible phototransformations. The photoresistance increases sharply on going from compound **1** with the thienyl moieties to derivative **17** with the benzothienyl moieties, which confirms the earlier advanced concept on a higher fatigue resistance of dihetarylethenes in which the bridges are linked with the condensed heterocycles or thiophenes with aryl

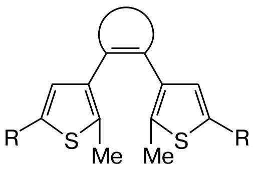
Table 1. Spectral kinetic characteristics of phototransformations of photochromic diarylethenes **1—21** in toluene

Compound	λ^{A}_{max}	λ^{B}_{max}	ΔD^{B}_{max}	k_{AB}/k_{BA}	$t_{0.5}$/s
	nm				
1	290	625	1.44	21.0	70
2	300	515	0.35	1.4	90
3	365	550	0.55	6.2	120
4	400	505	0.26	5.2	20
5	<300	450	0.41	1.1	25
6	290	455	0.46	1.9	8
7	240, 300	>350 (sh)	—*	—*	28
8	300	515	0.30	0.7	105
9	325	585	1.2	1.4	70
10	<300	470	0.28	2.1	>300
11	<300	420, 470	0.68	2.5	100
12	290	535	1.8	23.0	15
13**	290	530	1.8	13.8	10
14**	315	550	0.96	6.2	10
15	375	580	0.5	4.5	32
16	375	595	0.7	5.6	55
17	330	555	1.10	1.0	1040
18	340	495	1.75	1.3	750
19	405	510	0.13	0.2	360
20	300	450	1.46	0.6	60
21	300	455	0.38	—	24

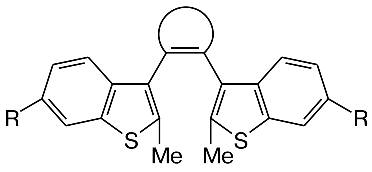
Note. Here and in Tables 2—5 λ^{A}_{max} and λ^{B}_{max} are the maxima of absorption bands of the opened (**A**) and cyclic (**B**) forms, respectively; ΔD^{B}_{max} is the maximum photoinduced absorbance in the absorption band maximum of the cyclic form **B** in the photoequilibrium state; k_{AB}/k_{BA} is the ratio of photocoloration and photodecoloration rate constants; $t_{0.5}$ is the time during which the absorbance achieved in the photostationary state at the absorption maximum of the cyclic form is halved upon continuous irradiation with the non-filtered light from a DRSh-250 lamp.

* Fast photodegradation.

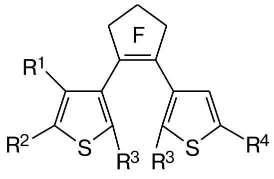
** In MeCN.



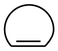
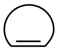
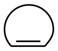
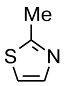
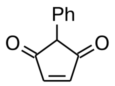
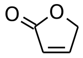
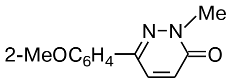
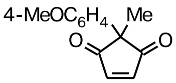
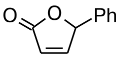
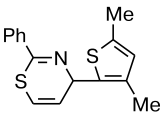
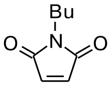
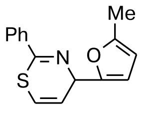
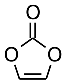
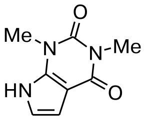
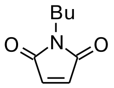
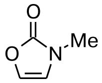
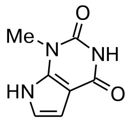
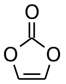
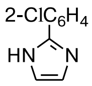
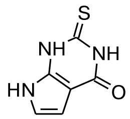
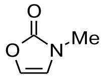
1—16

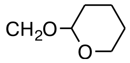
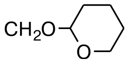
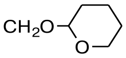


17—21



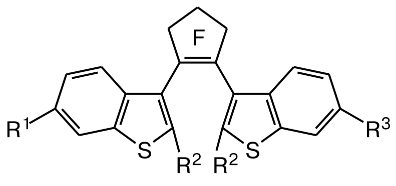
22—28

Compound		R	Compound		R	Compound		R
1		COH	**8**		Me	**15**		Me
2		Me	**9**		Me	**16**		Me
3		Me	**10**		Me	**17**		COH
4		Me	**11**		Me	**18**		COH
5		Me	**12**		Me	**19**		H
6		Me	**13**		Me	**20**		H
7		Me	**14**		Me	**21**		H

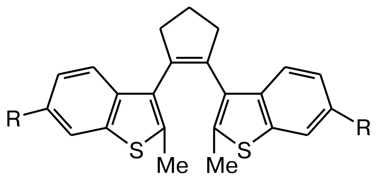
Compound	R1	R2	R3	R4	Compound	R1	R2	R3	R4	Compound	R1	R2	R3	R4
22	H	Me	Me	COH	**25**	H	CH_2OH	Me	CH_2OH	**27**	H		Me	
23	COH	Me	Me	COH	**26**	H	CH_2OH	Me		**28**	H	SEt	Et	SEt
24	H	COH	Me	CH_2OH										

or hetaryl substituents.[12] The spectral characteristics of the opened and cyclic forms of compound **17** are shown in Fig. 2. The replacement of the thienyl moieties by the benzothienyl ones results in the bathochromic shift of the absorption band of the opened form and the hypsochromic shift of the absorption band of the cyclic form, as well as an increase in the efficiency of the photodecoloration process of the latter.

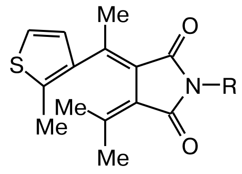
Depending on the structure of perfluorocyclopentene derivatives of diarylethenes, the absorption band maxi-



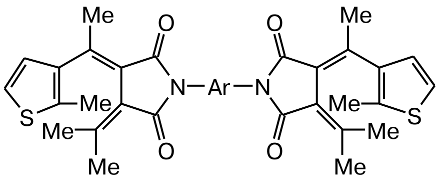
29—33



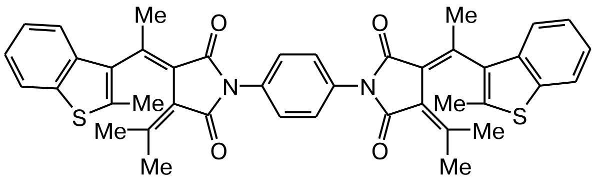
34—39



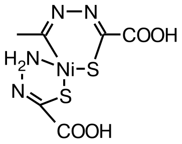
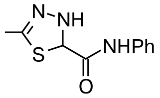
40—44

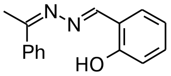
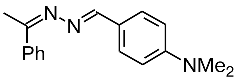
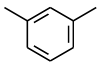
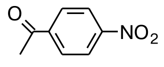
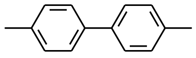
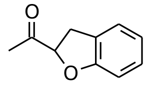
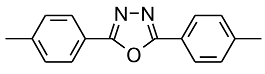


45—48



49

Compound	R^1	R^2	R^3	Compound	R^1	R^2	R^3	Compound	R^1	R^2	R^3
29	H	CH_2OMe	H	**31**	NO_2	CH_2OMe	NO_2	**33**	H	Me	
30	H	Me	COH	**32**	H	H					

Compound	R	Compound	R	Compound	R	Compound	Ar
34	CH(OH)Ph	**38**		**42**	$NHCO_2Bu$	**45**	
35	C(O)Ph	**39**		**43**	NPhCl	**46**	
36		**40**	Ph	**44**	$NPhNO_2$	**47**	
37		**41**	NH_2			**48**	

mum of the cyclic form can vary in a wide range: from 515 to 580 nm (Table 2). These compounds are characterized by a comparable sensitivity to the activating UV radiation and an appropriate photoresistance. In this case, the efficiency of photocoloration insignificantly exceeds the efficiency of photodecoloration.

The spectral kinetic study of the benzothienyl derivatives of diarylethenes (Table 3) shows that the absorption band maxima of their opened form exhibit the hypsochromic shifts compared to the thienyl derivatives. The introduction of substituents of different nature into the benzothienyl moieties exerts virtually no effect on the position of the absorption band maximum of the cyclic form. The photosensitivity and the ratio of the photocoloration and photodecoloration rate constants for the majority of substances are comparable with the corresponding parameters for the thienyl derivatives. The exceptions are compounds **32** and **33** with the bulky substituents, which are characterized by lower photosensitivity and efficiency of the photocoloration reactions compared to those of the photodecoloration process. All compounds of this type are more photosensitive than the thienyl derivatives of diarylethenes.

Cyclopentene derivative **18** is also characterized by an appropriate photoresistance (see Table 1). Its parameters determining the photosensitivity and efficiency of

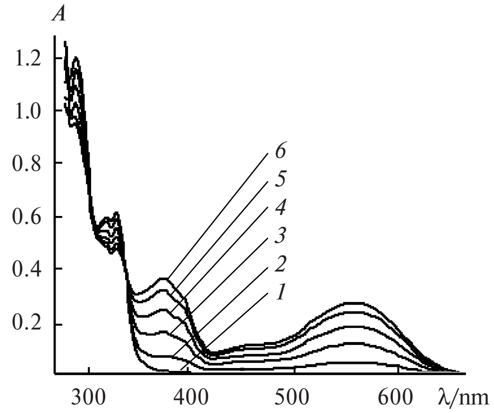


Fig. 2. Absorption spectra of perfluorocyclopentene diarylethene **17** in toluene before (*1*) and after consecutive UV-irradiation with the light at the wavelength 313 nm (*2—6*).

photocoloration and photodecoloration processes are comparable with those for perfluorocyclopentene analog **17**. The sharp difference is observed in the position of the absorption band of the cyclic form of this compound, whose absorption maximum exhibits a short-wavelength shift by 60 nm (Fig. 3).

The spectral kinetic study of other cyclopentene derivatives (Table 4) shows that the photochromic properties of these compounds are determined, to a substantial extent, by their structure. The replacement of the hydroxy-containing substituent in compound **34** by the carbonyl-containing substituent (compounds **35** and **36**) induces the bathochromic shift of the absorption bands of both opened and cyclic forms. Compound **35** manifests the highest photosensitivity. The introduction of nitro groups into the benzene fragments (compound **36**) decreases the photosensitivity and somewhat increases the photoresistance. Compound **37** turned out to be least photostable.

Unlike the above considered compounds, substances **38** and **39** are characterized by the low efficiency of photochromic transformations, which can be due to the *cis—trans*-photoisomerization of the azine fragments.

Table 2. Spectral kinetic characteristics of phototransformations of thienyl perfluorocyclopentene diarylethenes **22—28** in toluene

Compound	λ^{A}_{max} / nm	λ^{B}_{max} / nm	ΔD^{B}_{max}	k_{AB}/k_{BA}	$t_{0.5}$/s
22	295	380, 580	1.15	2.1	70
23	290	385, 565	0.76	2.4	90
24	290	365, 380, 580	1.40	3.3	80
25	300 (sh)	345, 515	0.98	2.5	160
26	285, 300 (sh)	345, 520	1.10	3.7	190
27	285	345, 525	0.65	3.6	220
28	<300	540	0.77	1.2	50

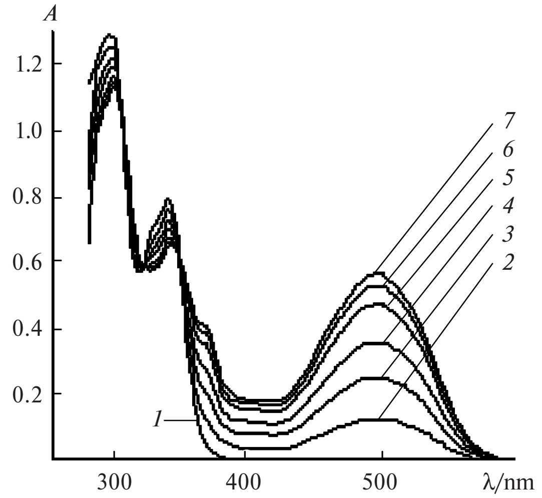


Fig. 3. Absorption spectra of cyclopentene diarylethene **18** in toluene before (*1*) and after consecutive UV-irradiation with the light at the wavelength 313 nm (*2—7*).

Benzothienyl maleimide derivative **19** possesses an increased photostability compared to thienyl-containing analog **4** (see Table 1). As compared to the perfluorocyclopentene and cyclopentene derivatives, compound **19** is characterized by a lower photosensitivity, a higher efficiency of photocoloration, and a sharp bathochromic shift of the absorption band of the opened form.

Compounds **3**, **8**, **10**, and **11** are also among photoresistant diarylethene derivatives (see Table 1).

For other compounds under study, as well as for those considered above, a stabilizing effect of the benzothienyl moieties compared to the thienyl fragments is pronounced (see Table 1, *cf.* compounds **5** and **20**, **6** and **21**). However, these compounds and other substances listed in Table 1 are inferior to the above considered diarylethenes in photostability and, hence, they have no special practical interest.

Since it is necessary to enhance the thermal stability of recorded optical information caused by the diffusion

Table 3. Spectral kinetic characteristics of phototransformations of benzothienyl perfluorocyclopentene diarylethenes **29—33** in toluene

Compound	λ^{A}_{max} / nm	λ^{B}_{max} / nm	ΔD^{B}_{max}	k_{AB}/k_{BA}	$t_{0.5}$/s
29	320	520	0.9	1.5	160
30	290, 330	365, 545	1.24	0.9	480
31	300, 340 (sh)	545	2.0	1.3	600
32	330	550	0.26	0.5	200
33	315	540	0.46	0.4	220

Table 4. Spectral kinetic characteristics of phototransformations of benzothienyl cyclopentene derivatives of diarylethenes **34—39** in toluene

Compound	$\lambda^{A}{}_{max}$	$\lambda^{B}{}_{max}$	$\Delta D^{B}{}_{max}$	k_{AB}/k_{BA}	$t_{0.5}$/s
	nm				
34	300	455	0.47	0.5	120
35	340	495	1.8	0.6	150
36	350	490	0.39	2.6	180
37	320	500	1.07	5.7	60
38	370	500	—	—	—
39	385	500	—	—	—

of photochromic molecules in the polymer layer and in order to increase the concentration of photosensitive components in the polymer layer, the photochromic polymers were prepared,[21,22] and among them product **50** was studied in most detail.

The spectral characteristics of the opened and cyclic forms of the photochromic fragment of this polymer are presented in Fig. 4.

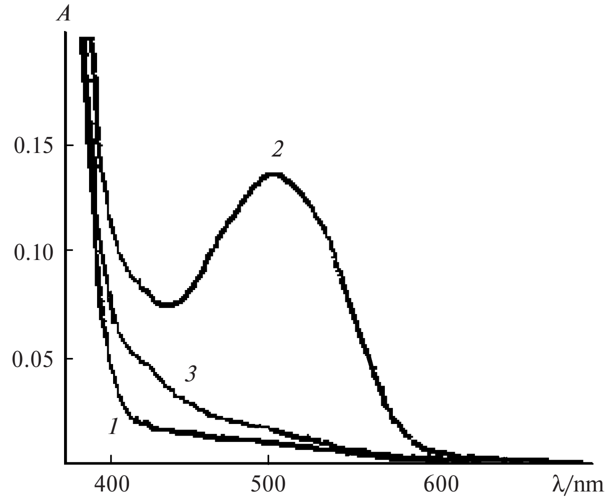


Fig. 4. Absorption spectra of photochromic polymer **50** before (*1*) and after consecutive irradiation with the UV (*2*) and visible (*3*) light.

Fulgimides. As in the case of diarylethenes, the study of the kinetics of thermal relaxation of photoinduced cyclic form **B** to the initial opened form **A** shows that the both forms are thermally stable for at least one month when storing the solutions in well closed vessels in dark.

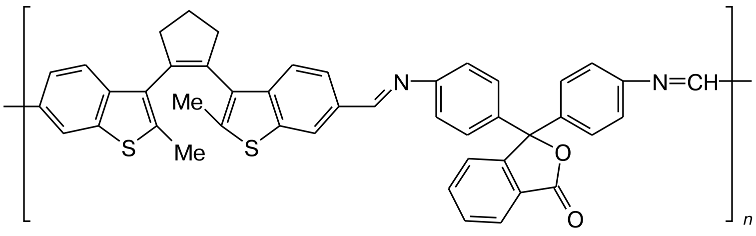
The spectral kinetic characteristics of the studied monofulgimides are presented in Table 5.

The absorption spectra of opened forms **A** of compounds **40—44** in toluene exhibit a band with a maximum at 290—330 nm. The absorption spectra of the initial and photoinduced forms of monofulgimide **41** irradiated with different doses of UV radiation are shown in Fig. 5. The series of the spectra contain an isosbestic point, indicating the absence of side processes in the mutual transformation of the both forms of this photochrome. It can be assumed that the *E—Z*-isomerization process typical of fulgides is inefficient in this case.

The results in Table 5 show that the position of the absorption band maximum of the opened form depends insignificantly on the structure of the compound, in particular, an increase in the electron-withdrawing properties of the R substituent results in a small hypsochromic spectral shift (*cf.* compound **40** and compounds **43** and **44**). Substances **40—42** are characterized by the longest-wavelength absorption bands of the opened form.

The cyclic forms of all the compounds absorb in the visible spectral region at 520—540 nm (see Fig. 5 and Table 5). However, unlike the opened form, the absorption band of the cyclic form shifts to the long-wavelength region compared to compound **40** with an increase in the electron-withdrawing properties (compounds **43** and **44**). Compounds **40** and **41** are characterized by the shortest-wavelength absorption of the cyclic form. An analysis of the kinetics of photocoloration of solutions of fulgimides in toluene (see Table 5) shows that the efficiency of this process decreases upon the introduction of electron-withdrawing substituents into the phenyl fragment (compounds **43** and **44**) with the simultaneous increase in the photoresistance of these compounds.

Synthesized bisfulgimides **45—48** are similar in their spectral kinetic properties to monofulgimides and characterized by the same absorption band maxima of both the opened and photoinduced forms (see Table 5). The exception is compound **48** for which the absorption band maximum of the opened form is shifted by 25 nm to the short-wavelength region. This means that the phenyl cycles and the heterocycle arranged between them are not



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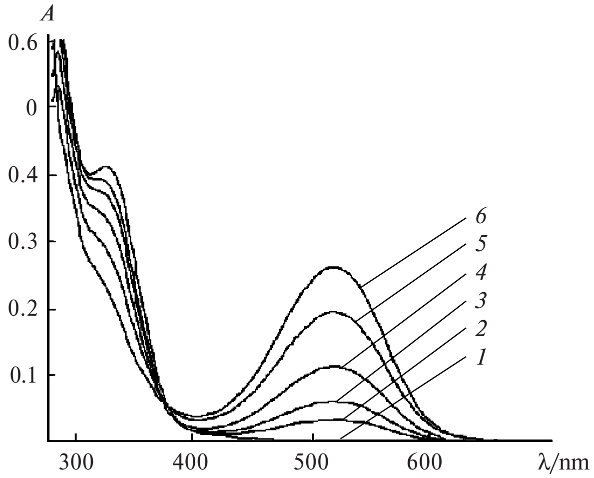

Fig. 5. Absorption spectra of monofulgimide **41** before (*1*) and after consecutive UV-irradiation with the light at the wavelength 313 nm (*2—6*).

virtually involved in the conjugation of two fulgimide residues.

The value of the photoinduced change in the absorbance at the wavelength of the absorption band maximum of the cyclic form shows that the photosensitivity of the compounds is virtually independent of the structure of the spacer between the fulgimide moieties.

Unlike photochromes **45—48**, the absorption band of the cyclic form of product **49** is shifted to the short-wavelength spectral region (by 32 nm compared to analog **45**) (Fig. 6, Table 5). The photodecoloration rate increases and the photoresistance of the compound is enhanced. By analogy to diarylethenes, it can be assumed that this occurs due to the replacement of the thienyl fragments by the benzothienyl ones.

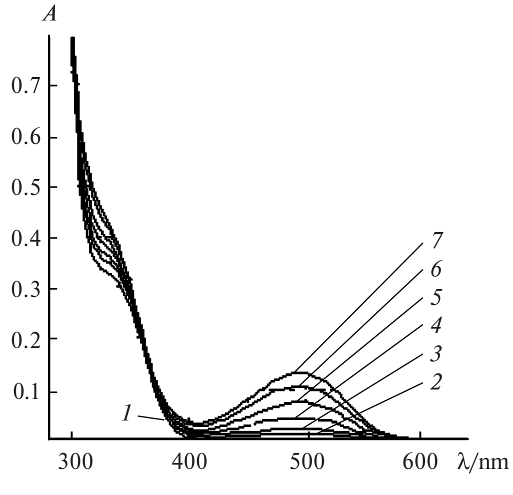

Fig. 6. Absorption spectra of bisfulgimide **45** before (*1*) and after consecutive UV-irradiation with the light at the wavelength 313 nm (*2—7*).

Table 5. Spectral kinetic characteristics of phototransformations of monofulgimides **40—44** and bisfulgimides **45—49** in toluene

Compound	λ^{A}_{max}	λ^{B}_{max}	ΔD^{B}_{max}	k_{AB}/k_{BA}	$t_{0.5}$/s
	nm				
40	250, 325	522	0.75	1.2	25
41	325	520	0.9	1.0	70
42	230, 280, 330	530	0.70	0.9	40
43	290	540	0.61	0.3	95
44	305	540	0.65	0.4	100
45	250, 330	528	1.2	0.8	30
46	285, 335 (sh)	530	1.5	0.4	50
47	285, 335 (sh)	530	1.5	0.4	50
48	310	530	1.1	0.4	50
49	335	495	0.75	0.1	220

An analysis of the presented results of the spectral kinetic study of thermally irreversible photochromic diarylethenes and fulgimides makes it possible to reveal the most promising directions of the synthesis of photochromic compounds that satisfy the requirements of using them as photosensitive components of photochromic detecting media for ODs of the three-dimensional bitwise operative optical memory of superlarge informational capacity. According to the whole body of the properties, the most promising for this application are perfluorocyclopentene and cyclopentene derivatives of diarylethenes. Photochromic fulgimides satisfy the requirements imposed on the spectral characteristics and photosensitivity but are inferior to the chosen diarylethenes in the fatigue resistance to photochromic transformations.

The samples of photochromic detecting media have been developed on the basis of the synthesized thermally irreversible photochromic perfluorocyclopentene diarylethenes and fulgimides that mostly satisfy the conditions of application.[32]

The revealed large diversity of spectral characteristics of the opened and cyclic forms of the synthesized diarylethenes allows the four-dimensional (4D) optical memory to be created, providing information recording and reproduction in different regions of the detecting medium volume and also at different wavelengths of laser radiation and, hence, leading to an increase in the informational capacity of ODs.

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